# FINAL PROGRAM

# Workshop on *In Situ* Methods in Nanomechanics

**August 1-3, 2007**

**Lawrence Berkeley National Laboratory**
**Berkeley, California, USA**

**Organizers**

**Andrew Minor (LBL) • Oden Warren (Hysitron, Inc.)**

**Sponsors**

Workshop Location: LBL, Berkeley, California (Building 66 Auditorium)
Workshop Website: http://ncem.lbl.gov/insituwks/in_situ_workshop_07.html
Workshop Email: in.situ.workshop@gmail.com
Workshop Reception and Workshop Hotel: http://www.claremontresort.com/
Workshop Banquet Dinner: http://www.skatesonthebay.com/

*View towards San Francisco Bay from Lawrence Berkeley National Laboratory*

## Workshop Focus

This **Workshop on *In Situ* Methods in Nanomechanics** aims to bring together researchers with an interest in combining nanomechanical (or closely related nanotribological) testing with real-time monitoring techniques such as transmission or scanning electron microscopy, x-ray diffraction, chemical spectroscopy, electrical current measurement, acoustic emission detection, *etc*. These emerging hybrid methods enable the building of direct relationships between, for example, microstructure evolution and distinct characteristics of force *vs*. displacement, or better yet, stress *vs*. strain curves. Such methods promise to improve our understanding of the origins of mechanical properties, and of how mechanical and other properties are coupled.

## Program Summary

The workshop begins with a JEOL-sponsored opening reception at The Claremont Resort & Spa, Berkeley from 7:00 PM to 8:30 PM on August 1, then proceeds to a full day of seminars followed by a poster session at Lawrence Berkeley National Laboratory on August 2, and concludes with a final day of seminars and a lab tour at LBL on August 3. Adding to a strong lineup of internationally recognized invited speakers, FEI Co. will provide an invited talk on its vision of the future of *in situ* methods. The opening reception, lunch at LBL on August 2 and 3, and a banquet dinner at Skates on the Bay restaurant, Berkeley Marina from 6:00 PM to 8:30 PM on August 2 are included with the workshop's registration fee. The Claremont Resort & Spa is the official hotel of the workshop.

**INVITED SPEAKERS**: William Nix (Stanford University) • Helena Van Swygenhoven (Paul Scherrer Institute, Switzerland) • Simon Ruffell (Australian National University) • John Balk (University of Kentucky) • Thomas LaGrange (Lawrence Livermore National Laboratory) • Daryl Chrzan (University of California, Berkeley) • Kathryn Wahl (US Naval Research Laboratory) • Aman Haque (Penn State University) • Jan Ringnalda (FEI Co.)

**POSTER PRIZE**: All workshop participants are encouraged to bring a poster. Posters do not have to strictly follow the workshop focus but should be of a subject matter that would be of interest to the workshop participants. The top poster as judged by the organizers will be awarded at the banquet dinner.

# Transportation and Presentation Information

## *Special shuttle bus service*

Special shuttle bus service has been arranged for all workshop events. The bus schedule is as follows:

Thursday August 2, 2007
8:30 AM – Main entrance of The Claremont Resort & Spa, Berkeley to Lawrence Berkeley National Lab, Bldg. 66
5:30 PM – LBL, Bldg. 72 to Skates on the Bay restaurant, Berkeley Marina
8:30 PM – Skates on the Bay to The Claremont Resort & Spa

Friday August 3, 2007
8:30 AM – Main entrance of The Claremont Resort & Spa to LBL, Bldg. 66
3:15 PM – LBL, Bldg. 66 to The Claremont Resort & Spa
4:30 PM – LBL, Bldg. 72 to The Claremont Resort & Spa

## *Other options for transportation to Lawrence Berkeley National Lab*

1) Regular LBL shuttle service (ideal for those taking BART):

LBL runs a regular shuttle service from downtown Berkeley to the Lab. Details can be found at:
http://www.lbl.gov/Workplace/Transportation.html#LBL-Shuttle

In order to board the buses all visitors must have a bus pass. To obtain a bus pass please request one prior to the workshop by emailing: in.situ.workshop@gmail.com with the subject line "Bus pass request".

2) Drive by car:

All visitors who are registered for the workshop can receive a parking permit for August 2nd and 3rd at either the Strawberry entrance or the Blackberry Canyon entrance to LBL. Please allow sufficient time (~15 minutes) to check-in at the gate and find a parking place if you choose to drive.

Directions to LBL can be found at:
http://www.lbl.gov/Workplace/Transportation.html

## *Directions to the Reception Site and the Official Workshop Hotel*

For those not utilizing the special shuttle bus service, directions to The Claremont Resort & Spa, Berkeley can be found at:
http://www.claremontresort.com/About/Directions/

## *Directions to the Banquet Dinner Site*

For those not utilizing the special shuttle bus service, directions to the banquet dinner at Skates on the Bay restaurant, Berkeley Marina can be found at:
http://www.skatesonthebay.com/maps.php

## *Oral Presentation Instructions*

An LCD projector, a laser pointer, and a sound system will be provided for the speakers. Speakers are expected to bring their own laptop computers for their oral presentations. Contact the organizers if this is not possible.

## *Poster Presentation Instructions*

Easels, 30”x40” foam board poster backing, double-sided poster tape, binder clips, and pushpins will be provided for poster presentations. If your poster is larger than 30”x40”, please bring it already mounted on backing.

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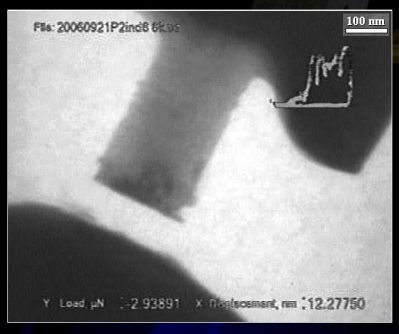
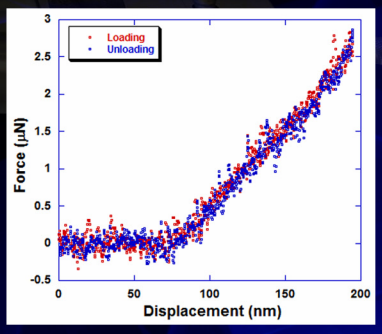
## nanopillar




**In-Situ Compression**

## nanowire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# Workshop Schedule

## *Day 1: Wednesday, August 1*

**Location: The Claremont Resort & Spa, Berkeley (Lanai 2)**

| **Time** | **Speaker/Event** |
|---|---|
| 7:00-8:30pm | Opening reception (sponsored by JEOL) |

## *Day 2: Thursday, August 2*

**Location: Lawrence Berkeley National Laboratory (Building 66 Auditorium)**

| **Time** | **Speaker/Event** |
|---|---|
| 8:30am | Bus departure from The Claremont Resort & Spa to the Workshop |
| 9:00-9:10 | Welcome address (by hosts, organizers) |

Early Morning Chair: Helena Van Swygenhoven, Paul Scherrer Institute

| | |
|---|---|
| 9:10-9:40 | **PLENARY:** William Nix, Stanford University<br>*The need for in situ observations of plastic deformation at the sub-micrometer scale* |
| 9:40-10:00 | Jack Houston, Sandia National Laboratories<br>*An in-situ SEM/IFM combination for studies of the mechanical properties of individual nanostructures* |
| 10:00-10:20 | Vikas Prakash, Case Western Reserve University<br>*In situ mechanical characterization of individual micro-/nano-scale fibers* |
| 10:20-10:40 | Break |

Late Morning Chair: Simon Ruffell, Australian National University

| | |
|---|---|
| 10:40-11:10 | **INVITED**: Daryl Chrzan, University of California, Berkeley and Lawrence Berkeley National Laboratory<br>*Novel materials deforming near their ideal strength* |
| 11:10-11:30 | Rod Ruoff, University of Texas at Austin<br>*Tensile loading known (n,m) SWCNTs and mechanics of 'graphene oxide paper'* |
| 11:30-11:50 | Jianyu Huang, Sandia National Laboratories<br>*In-situ plastic deformation of carbon nanotubes* |

11:50-12:10 Syed Asif, Hysitron, Inc.
*The role of surface forces and tip-surface interaction on the onset of plasticity*

12:10-1:00pm Working lunch

Early Afternoon Chair: Thomas LaGrange, Lawrence Livermore National Laboratory

1:00-1:30 **INVITED:** Aman Haque, Penn State University
*MEMS-based tools for in-situ nanomechanical testing*

1:30-1:50 Scott Mao, University of Pittsburgh
*In-situ TEM study on deformation and fracture of nanocrystalline materials*

1:50-2:10 Nathan Mara, Los Alamos National Laboratory
*In-situ observation of superplasticity and cooperative grain boundary sliding in nanocrystalline $Ni_3Al$*

2:10-2:30 Donna Ebenstein, Bucknell University
*Correlating nanomechanical properties with chemical composition and surface morphology in silk films using micro-Raman spectroscopy and stiffness imaging*

2:30-2:50 Break

Late Afternoon Chair: John Balk, University of Kentucky

2:50-3:20 **INVITED:** Kathryn Wahl, US Naval Research Laboratory
*In situ tribology: What's really happening in the buried sliding interface?*

3:20-3:40 Daan Hein Alsem, Lawrence Berkeley National Laboratory
*Nanoscale tribology of polycrystalline silicon structural films*

3:40-4:00 Laurence Marks, Northwestern University
*Friction in Full View*

4:00-4:30 **INVITED:** Jan Ringnalda, FEI Co.
*New developments in aberration corrected S/TEM microscopy: A new era for in-situ structure-property relationships studies*

4:30-5:30 Poster session (National Center for Electron Microscopy, Building 72)

5:30 Bus departure from Building 72 to Skates on the Bay, Berkeley Marina

6:00-8:30 Banquet dinner at Skates on the Bay, Berkeley Marina

8:30 Bus departure from Skates on the Bay, Berkeley Marina to The Claremont Resort & Spa

## Day 3: Friday, August 3

**Location: Lawrence Berkeley National Laboratory (Building 66 Auditorium)**

| **Time** | **Speaker/Event** |
|---|---|
| 8:30am | Bus departure from The Claremont Resort & Spa to the Workshop |

Early Morning Chair: Kathryn Wahl, US Naval Research Laboratory

9:00-9:30 **KEYNOTE:** Helena Van Swygenhoven, Paul Scherrer Institute
*In-situ micro-compression in the Swiss Light Source*

9:30-9:50 Zhiwei Shan, Hysitron, Inc.
*Perfecting nanostructural single crystal Ni through stress/strain annealing*

9:50-10:10 Jia Ye, Lawrence Berkeley National Laboratory
*Quantitative in-situ TEM nano-compression tests of AA6063 aluminum alloys*

10:10-10:30 Break

Late Morning Chair: Aman Haque, Penn State University

10:30-11:00 **INVITED:** John Balk, University of Kentucky
*In situ observations of deformation during indentation of nanoporous gold thin films*

11:00-11:20 Takahito Ohmura, National Institute for Materials Science
*Observation of dislocation-grain boundary interactions in martensitic steel through in-situ nanoindentation in a TEM*

11:20-11:40 Lars Johnson, Linköping University
*In situ TEM nanoindentation studies of alpha-$Al_2O_3$ and $Ti_3SiC_2$*

11:40-12:00 Michel Barsoum, Drexel University
*On the determination of spherical nanoindentation stress-strain curves and their importance*

12:00-1:00pm Working lunch

## Afternoon Chair: Daryl Chrzan, University of California, Berkeley

| | |
|---|---|
| 1:00-1:30 | **INVITED:** Simon Ruffell, Australian National University<br>*In-situ electrical characterization during nanoindentation in silicon* |
| 1:30-1:50 | Dylan Morris, National Institute of Standards and Technology<br>*Multi-scale measurement of contact forces and current with a custom adhesion apparatus* |
| 1:50-2:10 | Ryan Major, Hysitron, Inc.<br>*Conductive nanoindentation: In-situ correlation of mechanical properties, deformation behavior, and electrical characteristics of materials* |
| 2:10-2:40 | **INVITED:** Thomas LaGrange, Lawrence Livermore National Laboratory<br>*Application of time-resolved transmission electron microscopy to in situ deformation studies* |
| 2:40-3:00 | Mitra Taheri, Lawrence Livermore National Laboratory<br>*An environmental stage for the dynamic TEM: In situ microstructural evolution in varied atmosphere at nanosecond scales* |
| 3:00-4:30 | Lab tour of the National Center for Electron Microscopy (Building 72) |
| 3:15 | Bus departure from Building 66 to The Claremont Resort & Spa for those unable to participate in the lab tour |
| 4:30 | Bus departure from Building 72 to The Claremont Resort & Spa for those able to participate in the lab tour |

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# ABSTRACTS – ORAL PRESENTATIONS

## Thursday, August 2 – Early Morning

## PLENARY 9:10-9:40

### The need for in situ observations of plastic deformation at the sub-micrometer scale

**W.D. Nix**
*Department of Materials Science and Engineering*
*Stanford University*

"Seeing is believing" and "A picture is worth a thousand words" are two of the most well accepted old sayings in our culture. It is natural for them to have an appeal in materials science as well. Here we review some recent experiments on plastic deformation of crystalline materials at the sub-micrometer scale that call for in situ observations of the controlling processes.

It is well known that thin films are much stronger that bulk materials. This is seen in measurements of the stresses in metal films on silicon substrates subjected to thermally cycling. The effects of film thickness are dramatic; pure gold films, 0.2 micrometers thick, can sustain stresses of nearly 500 MPa at 600˚C. For many years the cause of this dramatic strengthening effect has been attributed to the confinement of dislocations by the thickness of the film and by related dislocation interactions that lead to extraordinary rates of strain hardening. But efforts to make direct, in situ, observations of these processes have not led to a clearer picture of thin film strength. While some evidence for dislocation storage (and strain hardening) is found, there is also evidence that plastic flow in very thin films might be limited by the availability of dislocation sources. Further work is needed.

When crystals are deformed by indentation using sharp indenters, the hardness frequently decreases with increasing depth of indentation. This has been explained by considering the geometry of indentation deformation and the geometrically necessary dislocations and strain gradients that must accompany indentation. According to this picture, the hardness increases with decreasing depth of indentation because the total length of geometrically necessary dislocations forced into the solid by the self-similar indenter scales with the square of the indentation depth, while the volume in which these dislocations are found scales with the cube of the indentation depth. This leads to a geometrically necessary dislocation density that depends inversely on the depth of indentation and an indentation size effect. Recent experiments designed to reveal the lattice curvatures associated with small indentations will be discussed. Other experiments involving lattice curvatures and geometrically necessary dislocations induced into metal interconnect lines will be described. All of these experiments would benefit from in situ TEM observations.

While the indentation size effect can be well explained using the idea of geometrically necessary dislocations, that explanation implies that small single crystals subjected to *uniform* deformation would not be stronger than bulk materials. But many recent experiments involving the compressive deformation of sub-micrometer pillars of various FCC metals show that a dramatic size effect is present even though there are no significant gradients of strain present. Recent ex-situ x-ray micro-diffraction studies of the structural state of gold pillars, before and after compressive deformation, show that strain hardening is not responsible for the observed size effect. Dislocation starvation, or source-controlled plasticity, has been suggested as the cause of this size effect. Recent in situ TEM observations of compressively deformed Ni pillars, by Minor et al., supports this basic idea.

While dislocation source starvation has been suggested only recently in connection with the strength of pillars and nano-crystalline metals, there remains the possibility that this strengthening mechanism might be operating in other situations where "smaller-is-stronger." In situ experiments may help to determine the role that this strengthening mechanism might play in other experiments.

## 9:40-10:00

### An In-Situ SEM/IFM Combination for Studies of the Mechanical Properties of Individual Nanostructures

**J. E. Houston and B. S. Swartzentruber**
*Sandia National Laboratories*
*Albuquerque, NM 87185*

In order to study the detailed mechanical properties of nanostructures it is necessary to have the ability to both controllably locate and manipulate individual structures and to quantitatively and stably measure the forces involved. In this presentation, we outline the development of an instrument consisting of a combination of field-emission scanning electron microscopy (SEM), interfacial force microscopy (IFM) and a nanometer-level metrology platform. This combination permits the quantitative measurement, for example, of elastic deformation, yield by the nucleation of individual dislocations or viscoelastic plastic flow. The SEM has < 2nm spatial resolution enabling it to locate and manipulate, as well as follow the displacement of, nanostructures. The manipulator is able to controllably make XYZ displacements at the sub-nm level. The IFM utilizes a unique self-balancing, force-feedback sensor, which eliminates the instability seen in all compliance-based sensors such as the atomic force microscope (AFM). Coupled with a dual laser interferometer arrangement, the sensor is capable of the independent measurement of forces in two dimensions over a range from the sub nN level up to about 0.5 mN. These capabilities will be demonstrated from earlier nanomechanics and nano-manipulation efforts.

**10:00-10:20**

## IN SITU MECHANICAL CHARACTERIZATION OF INDIVIDUAL MICRO-/NANO-SCALE FIBERS

**Pankaj Kaul and Vikas Prakash**
*Department of Mechanical and Aerospace Engineering,*
*Case Western Reserve University*
*Cleveland, OH 44106*

We report the development of a novel test and characterization device for obtaining the mechanical behavior of one-dimensional nanostructures. The device features independent measurements of both the load and displacement history in micro-/nano- scale specimens with micro-Newton force and nanometer-scale displacement resolutions, respectively. Moreover, the tool is well suited for *in-situ* testing within a SEM, which permits continuous high-resolution imaging during the nanomechanical straining. The device comprises of two main parts: (a) a three-plate capacitive transducer (Hysitron, Inc) that doubles-up both as an actuator and a force sensor; and (b) a nanomanipulator (Kliendiek MM3A) that facilitates transportation and positioning of the nanoscale structures with nanoprecision. To conduct the experiments, the ends of the specimen are individually attached to the probe tips on both the nanomanipulator and the transducer using nanopositioning and ion and/or electron-beam induced deposition. This process involves precision metering of pre-determined organo-metallic gas precursors into the specimen chamber via a micro-delivery gas-injection system, where the high intensity (electron or ion) beam is used to form relatively high-strength platinum deposits at its focal point. In our present study, FEI's Platinum Deposition system (Model: FP 3400/30) was employed to provide a source for Pt deposition. The workings and capabilities of the testing device are illustrated by presenting results of nanomechanical characterization of micron-sized diameter polyaniline fibers.

# Thursday, August 2 – Late Morning

## INVITED 10:40-11:10

### Novel Materials Deforming Near Their Ideal Strength

**Daryl C. Chrzan**
*Materials Science and Engineering, University of California, Berkeley, California, and*
*Materials Sciences Division, Lawrence Berkeley National Laboratory, Berkeley, California*

Nanoindentation within a transmission electron microscope provides an excellent opportunity for probing the strength of structures at the nanoscale, and for comparing the observed response with the predictions of theory. This talk considers the mechanical response of two novel materials. First, compression tests of CdS nanospheres are considered. Experimental results are compared with predictions of a finite element analysis to show that the CdS nanospheres are deforming at stresses approaching their ideal strength. Second, the nanoindentation deformation of Ti-based Gum Metal alloys is studied. These alloys are the first known to deform in bulk form at or near their ideal strength without the apparent motion of dislocations. The results of these experiments are interpreted within a newly developed theory that explains the mechanical response of these unusual alloys.

This work is supported by the Department of Energy, Toyota, and the National Science Foundation.

## 11:10-11:30

### Tensile loading known (n,m) SWCNTs and mechanics of `graphene oxide paper´

**Rod Ruoff**
*Department of Mechanical Engineering*
*Northwestern University (in transition to UT Austin, Fall '07)*

I provide a brief update on fracture mechanics of SWCNTs of known diameter and chirality. I then turn to discussing ***graphene-based materials***. Our top-down approaches [1] directly served to propel physicists to study individual layers of graphite but our current approach has been to convert graphite to graphite oxide (GO), generate colloidal suspensions of individual layers of GO in water, and to use these individual layers in a variety of ways. For example, we have embedded individual and reduced 'graphene oxide' sheets in polymers such as polystyrene and evaluated their dispersion, sheet morphology, and the electrical percolation and conductivity of the resulting composites. In parallel paths, we have (i) undertaken studies of individual graphene oxide and reduced graphene oxide sheets, to elucidate their optical and electrical properties, (ii) embedded graphene oxide sheets in glass by a sol-gel route thereby making electrically conductive and transparent glass coatings, and (iii) produced 'graphene

oxide paper', a material with intriguing mechanical properties. In line with this workshop, I will focus primarily on topic (iii).

1. Lu XK, Yu MF, Huang H, and Ruoff RS, *Tailoring graphite with the goal of achieving single sheets,* Nanotechnology, **10**, 269-272 (1999).

Support of our work by the NSF, ONR/NRL, NASA, and DARPA is appreciated.

## 11:30-11:50

### In-Situ Plastic Deformation of Carbon Nanotubes

**Jianyu Huang**
*Sandia National Laboratories*
*Center for Integrated Nanotechnologies (CINT)*
*Dept. 01132, P.O. Box 5800, MS 1303, Bldg 518*
*Albuquerque, NM 87185*

Currently there exists a gap between the microstructure and the corresponding electrical and mechanical property studies of nanostructured materials, i.e. studying the microstructure without knowledge of the related physical properties, or vice versa. By using a Nanofactory transmission electron microscopy-scanning tunneling microscopy (TEM-STM) platform, we were able to characterize simultaneously the atomic-scale microstructure with its electrical and mechanical properties of individual carbon nanotubes [1-3], which are perceived as being rather brittle because of the strong C-C $sp^2$ bonds in the honeycomb lattice. It is postulated that nanotubes accommodate no *plastic* deformation even beyond the elastic limit or before breakage at room temperatures. I report here our recent discoveries of plastic deformation, as characterized by the superplastic elongation, kink motion, and dislocation climb, in carbon nanotubes at about 2000 °C. The plastic deformation induces dramatic electronic property changes of the nanotubes. These discoveries indicate that there are rich nanomechanics and nanoelectronics in carbon nanotubes at high temperatures. Our discoveries may provide important implications for the high temperature applications of carbon nanotubes.

[1] J. Y. Huang *et al*. Nature 439, 281 (2006).
[2] J. Y. Huang *et al*., Phys. Rev. Lett. 94, 236802 (2005); 97, 075501 (2006); 98, 185501 (2007).
[3] J.Y. Huang *et al*., Nano. Lett. 6, 1699 (2006).

## 11:50-12:10

### The role of surface forces and tip-surface interaction on the onset of plasticity

**S.A. Syed Asif[1], Oden L. Warren[1], Zhiwei Shan[1,2], Andrew M. Minor[2]**
[1]*Hysitron, Incorporated, Minneapolis, MN*
[2]*National Center for Electron Microscopy, Lawrence Berkeley National Laboratory, Berkeley, CA*

The quantitative study of the mechanical properties of materials at the nanoscale has been receiving much attention in recent years. For submicron-scale mechanical property measurement, depth-sensing nanoindentation techniques are very successful and gaining popularity. However, below the 10nm length scale it is difficult to understand and model the material behavior using depth-sensing indentation alone. At this scale the tip-surface interaction forces and the surface chemistry are very important and it is extremely difficult to correlate the mechanical response measured in the form of load-displacement curves to the actual deformation behavior. Combining the force modulation technique with depth sensing improves the sensitivity of the measurement and avoids problems like thermal drift; however, this does not help to correlate the modes of deformation to the measurement. In this presentation we show that the in-situ TEM based, displacement controlled, depth-sensing indentation technique helps to correlate the tip-surface interaction to the deformation behavior directly. The role of surface forces, surface oxide layer, and adhesion on the earliest stage of plasticity will be demonstrated using a diamond indenter tip interacting with an aluminum surface with a native oxide layer covering the surface.

# Thursday, August 2 – Early Afternoon

## INVITED 1:00-1:30

### MEMS-based Tools for In-situ Nanomechanical Testing

**Aman Haque**
*The Pennsylvania State University*

In-situ testing of nanoscale materials in SEM/TEM or other analytical tools is the only way one can obtain quantitative (strain-strain) and qualitative (dislocation, crack visualization) simultaneously, with the added benefit of monitoring 'cleanliness' of the experiments. Unfortunately, these environments have serious space restrictions; a concern we address using nanofabrication techniques. In this talk, we will present three examples of custom-designed nanofabricated tools compatible with virtually any form of microscopy. The first example involves force/displacement resolution on the order of 100 nano-Newtons and 100 nanometers respectively and is suitable for uni-axial tensile testing of freestanding thin films or microtomed TEM specimens. We will present in-situ TEM and SEM experimental results on 30-100 nm thick metal films. The second example involves same capabilities with better resolutions (100 pico-Newtons and 1 nm) that make it suitable for individual nanotubes and nanowires. We will present experimental results on Zn) nanowires. The third example is on normal/friction force measurement with about 1 nano-Newton force resolution.

## 1:30-1:50

### In-situ TEM study on deformation and fracture of nanocrystalline materials

**Scott X. Mao[1], Zhiwei Shan[1], J.M.K Wiezorek[2], J.A. Knapp[3], D.M. Follstaedt[3] and E.A. Stach[4]**
[1]*Department of Mechanical Engineering, University of Pittsburgh, Pittsburgh, PA 15261, USA*
[2]*Department of Materials Science and Engineering, University of Pittsburgh, Pittsburgh, PA 15261, USA*
[3]*Sandia National Laboratories, Albuquerque, NM 87185, USA*
[4]*School of Materials Engineering, Purdue University, West Lafayette, IN 47907, USA*

In general, the plastic behavior of crystalline materials is mainly controlled by the nucleation and motion of lattice dislocations. We used in situ dynamic transmission electron microscopy to observe nanocrystalline nickel with an average grain size of about 10 nanometers, which shows deformation-induced grain agglomeration. It has been found that grain boundary mediated processes have become a prominent deformation mode. Additionally, trapped lattice dislocations are observed in individual grains following deformation. This change in the deformation mode arises from the grain-size dependent competition between the deformation

controlled by nucleation and motion of dislocations and the deformation controlled by diffusion assisted grain boundary processes.

Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

## 1:50-2:10

### In-situ Observation of Superplasticity and Cooperative Grain Boundary Sliding in Nanocrystalline $Ni_3Al$

**N.A. Mara*, A.V. Sergueeva, A.K. Mukherjee**
**Present address: Los Alamos National Laboratory*

Cooperative grain boundary sliding (CGBS) has shown to account for the majority of macroscopic strain seen in microcrystalline metallic systems undergoing superplastic deformation. While CGBS has been observed on the surface of microcrystalline samples deforming superplastically through the shifting of diamond scribe lines, past work has not included TEM results showing occurrence in the bulk of the material, and the details behind the micromechanism of CGBS. In this work, nanocrystalline $Ni_3Al$ produced via High Pressure Torsion is deformed superplastically in the TEM. High-temperature (~700 C) in-situ tensile testing shows the nature of CGBS at the nanoscale through direct observation of this phenomenon.

## 2:10-2:30

### Correlating Nanomechanical Properties with Chemical Composition and Surface Morphology in Silk Films Using Micro-Raman Spectroscopy and Stiffness Imaging

**Donna M. Ebenstein**
*Biomedical Engineering Program*
*Bucknell University*
*Lewisburg, PA 17837*

Relationships between structure and function are the key to understanding the behavior of biological materials and to developing tissue replacements and other biomimetic materials. By combining nanomechanical testing with spatially resolved chemical and structural analysis techniques, structure-property relationships at the nano- or microscale level can be investigated in diverse biomaterials. To demonstrate the utility of combining these techniques, I will share a study of silkworm silk films that were processed to induce different secondary structures in the silk fibroin protein. Research has shown that certain sequences of silk fibroin protein can form

two distinct structures: a less structurally organized silk I phase and a silk II phase made up of more highly structured beta-sheets. In this study, films made from silkworm fibroin protein under a variety of different processing techniques, including methanol, water, stretching, and surface etching treatments. Quasi-static nanoindentation was used to measure film moduli, dynamic stiffness imaging using the nanoindenter provided high resolution images of film surface morphology, and micro-Raman spectroscopy was used to characterize the predominate protein conformation in each film. Combining the information from all three techniques allowed interpretation of the complex relationship between protein conformation, film microstructure and the nanomechanical properties of silk films.

# Thursday, August 2 – Late Afternoon

## INVITED 2:50-3:20

### *In situ* tribology: What's really happening in the buried sliding interface?

**Kathryn J. Wahl**
*Tribology Section, Code 6176*
*U.S. Naval Research Laboratory*
*Washington DC 20375-5342*

Tribological processes that influence friction and wear involve a complex combination of materials science, physics, chemistry, and rheology. Our understanding of these sliding contact phenomena is limited by the fact that all the action takes place in a buried interface. Most often the only evaluation of these interfaces is accomplished through *ex situ* means after separating the contacts. *In situ* approaches to studying friction and wear processes are challenging because most engineering surfaces are metals or ceramics that have no optical transparency at visible wavelengths. For this reason, most of what is known about interfacial processes occurring during sliding has been learned through optical probes of sliding interfaces. Furthermore, as we work towards understanding interfacial phenomena in smaller, sub-optical scale contacts, *in situ* methods become increasingly challenging to implement.

In this talk, I will present examples of the kinds of physical and chemical processes occurring in buried sliding interfaces. Film thickness, chemistry/phase, rheology, morphology, and contact pressure are readily determined by *in situ* optical methods. These real-time, *in situ* methods show a rich variety of materials processing phenomena occurring during sliding. *In situ* experiments have demonstrated that steady state friction values often correlate with interface chemistry, while dynamic instabilities are associated with morphology and interfacial film thickness changes. I will give examples of how *in situ* measurements have helped us understand a variety of tribology phenomena. I will conclude by outlining scientific issues and opportunities for advancing our understanding of interfacial phenomena in tribology, especially as applied to micro- and nanoscale contacts.

## 3:20-3:40

### NANOSCALE TRIBOLOGY OF POLYCRYSTALLINE SILICON STRUCTURAL FILMS

**D.H. Alsem[1,2], E.A. Stach[3], M.T. Dugger[4] and R.O. Ritchie[2,5]**
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Tribological properties associated with friction and wear are critical factors in the reliability of silicon nano- and micromicroelectromechanical systems (NEMS/MEMS). Specifically, the generation of nanoscale wear particles and changing frictional behavior during operation is potentially catastrophic for applications where debris or an increase in friction may inhibit motion. Accordingly, it has become important to fully understand the nanomechanical processes associated with wear and friction in polysilicon structural thin films. To address this issue, we have run sidewall friction and wear MEMS test devices (fabricated in the Sandia SUMMiT™ process) in ambient air. Analytical scanning and transmission electron microscopy was used to study surface morphology and microstructural evolution of nanoscale wear debris and worn polysilicon. Values of the static coefficients of friction as a function of number of wear cycles were also determined and related to the evolution of the surface morphology and roughness. Atomic force microscopy was utilized to quantify the volume of nanoscale wear as function of number of cycles. All these measurements point to a dominating three-body abrasive wear mechanism, governed by nanoscale debris particles created by fracture. A custom built *in situ* TEM biasing holder is currently being developed to gain further insight in these nanoscale wear processes.

## 3:40-4:00

### Friction in Full View

**A. Merkle and L. D. Marks**
*Department of Materials Science and Engineering*
*Northwestern University*
*Evanston, IL 60208*

Experiments in tribology have long suffered from the inability to directly observe what takes place at a sliding contact - the classic buried interface problem. As a consequence, although many friction phenomena at the nanoscale have identified, there can be interpretation issues resulting from indirect or ex-situ characterization of the contact surfaces or because the

experimental measurements are volume averaged, rather than giving direct insight into what is taking place at a single asperity-asperity contact. We have been recently exploiting a unique instrument that allows us to simultaneously slide a tip across a surface and look at the sample using transmission electron microscopy. Using this technique, we can directly image the nanoscale processes taking place at scales from 0.2 nm to microns, as well as obtain local chemical information from techniques such as electron energy loss spectroscopy. Using this instrument we have recently observed "liquid-like" deformation where the material is solid, but behaves as if it was a liquid due to very rapid surface diffusion, similar to the classic case of liquid-like growth of gold and silver particles; the formation of a graphitic transfer layer during sliding of tungsten on graphite as well as in-situ observation of graphitization of diamond-like carbon during sliding observed by electron energy loss spectroscopy. Further results include observation of wear debris during sliding of tungsten on graphite whose size is consistent with a dislocation standoff model and a recently published dislocation model for friction at the nanoscale. These and additional results will be described.

## INVITED 4:00-4:30

### New Developments in Aberration Corrected S/TEM Microscopy: A New Era for In-Situ Structure-Property Relationships Studies

**Jan Ringnalda, Bert Freitag, and Dominique H.W. Hubert**
*FEI Company, PO Box 80066, 5600 KA Eindhoven, The Netherlands*

As the limits of nanotechnology are expanded ever further, so too must we push back the frontiers of imaging and analysis. The need for tools that can deliver ultra-high resolution information is driving the development of electron microscopy and spectroscopy to the extremes of performance. For example, aberration-corrected S/TEM gives us the ability to work at sub-angstrom length-scales. This, combined with sharply-defined energy resolution, gives us the capability to acquire information at the single atomic level and gain knowledge of inter-atomic bonding, to enable characterisation of chemical composition, electronic structure and mechanical properties. In addition, there is scope for capturing time-resolved structural transformations with sub-nanometer detail, enabling us to directly observe and understand the dynamics of a range of chemical processes *in situ*.

The capability of directly interpretable images at atomic resolution promises to revolutionise materials science. It crosses an important threshold in allowing researchers to investigate material properties in terms of individual atomic and molecular mechanisms rather than as the bulk properties of an aggregate population. In a practical sense, ease of operation and interpretation provides access to new information and new results, allowing scientists to spend their time and effort applying results rather than obtaining them — and seeing things that have never been seen before. Equally important, corrected S/TEM allows breaking the traditional relationship between ultimate resolution and sample space within the objective lens (see also:

http://www.lbl.gov/LBL-Programs/TEAM/index.html The US Department of Energy's Office of Science TEAM Project). Now, ultimate sub-Angstrom direct imaging is possible without sacrificing the space for the sample (pole piece gaps of 5mm+): "Space to do more". This larger space will certainly prove beneficial for new hybrid and correlative analysis at the ultimate level: combining ultimate S/TEM with in-situ mechanical measurement technologies.

# Friday, August 3 – Early Morning

## KEYNOTE 9:00-9:30

### In-situ micro-compression in the Swiss Light Source

**Helena Van Swygenhoven**
*Paul Scherrer Institute, CH-5232 Villigen-PSI, Switzerland*

To address small scale plasticity in object size confined structures, real time resolved in-situ white-beam Laue microdiffraction experiments are performed at the MicroXAS beamline of the Swiss Light Source (SLS). A micro-compression unit, equipped with a Hysitron TriboScope® single axis transducer, generating both load and displacement via the applied voltage on the three-plate capacitive transducer, is used to deform the pillars. Polychromatic diffraction patterns were obtained using conventional Laue transmission geometry with photon energies ranging from 2 keV to 24 keV. Kirkpatrick-Baez mirror focusing optics was used to obtain a beam FWHM of 1.5 to 2.5 μm2 in the focal plane with a maximum angular divergence of 0.2 x 0.3 mrads.

The dynamics of the Laue patterns of Au pillars demonstrate the occurrence of crystal rotation during compression and underline the role of the pillar's initial microstructure. The strengthening in the smaller pillar can be explained by plasticity starting on a slip system that is geometrically not predicted but selected because of the character of the pre-existing strain gradient. These results underline the importance of in-situ techniques, shed light on the dynamics of small scale plasticity and disclose the significance of the experimental boundary conditions when developing a theoretical or computational framework for fundamental understanding of small-scale plasticity.

## 9:30-9:50

### PERFECTING NANOSTRUCTURAL SINGLE CRYSTAL NI THROUGH STRESS/STRAIN ANNEALING

**Z. W. Shan[1,2], Raj Mishra[3], S.A. Syed Asif[1], O. L. Warren[1] and A. M. Minor[2]**
[1]*Hysitron Incorporated, 10025 Valley View Road, Minneapolis, Minnesota 55344, USA*
[2]*National Center for Electron Microscopy, Lawrence Berkeley National Laboratory, Berkeley, California 94720, USA*
[3]*Motors Research and Development Center, Warren, Michigan 48090, USA*

Dislocations that are close to free surfaces are known to be attracted to the surface by what so called image force. We report compression tests on submicron-diameter, single-crystal Ni pillars performed inside a transmission electron microscope (TEM). It was found that pillars fabricated

with a focused ion beam (FIB) initially possess a high defect density, but under compression the detect density can be dramatically reduced and can even leave behind a pillar completely free of dislocations. Analysis indicates this is due to image force effects. This phenomenon, denoted "stress/strain annealing", not only provides compelling direct experimental evidence for interpreting the unusual mechanical behavior of small volumes, but also raises the expectation of generating microstructurally perfect nanostructures by eliminating defects through stress/strain annealing.

## 9:50-10:10

### Quantitative in-situ TEM nano-compression tests of AA6063 aluminum alloys

**J. Ye[1], R. Mishra[2], A.M. Minor[1]**

[1]*National Center for Electron Microscopy, Lawrence Berkeley National Laboratory, Berkeley, CA 94720*

[2]*General Motors Research and Development Center, Warren, MI 48090*

We have studied the mechanical properties and plastic deformation mechanisms of AA6063 aluminum alloys through quantitative in-situ nano-compression tests in a transmission electron microscope (TEM). Two AA6063 alloys with identical compositions were studied for comparison, one as-extruded and the other heat treated to increase the solute concentrations in the matrix. By performing in-situ nano-compression tests on focused ion beam (FIB) fabricated submicron pillars, we found that the treated sample exhibits a higher yield stress than the other sample. In addition, dynamic observation and comparison of the two alloys demonstrated that the deformation mechanisms are quite different for the two samples. The treated sample showed a complex three-dimensional deformation through limited dislocation sliding, as compared to the simple two-dimensional slip of the as-extruded sample. These results demonstrate the dramatic effect of solutes on the plastic deformation behavior in AA6063 alloys and will help us better understand the fundamental mechanisms of plastic deformation in Al alloys.

# Friday, August 3 – Late Morning

## INVITED 10:30-11:00

### *In situ* Observations of Deformation During Indentation of Nanoporous Gold Thin Films

**Ye Sun[1], Jia Ye[2], Zhiwei Shan[2,3], Andrew M. Minor[2] and T. John Balk[1]**
[1]*University of Kentucky, Chemical and Materials Engineering, Lexington, KY*
[2]*National Center for Electron Microscopy, Berkeley, CA*
[3]*Hysitron Inc., Minneapolis, MN*

Thin films of nanoporous gold (np-Au) consist of open pores and ligaments, the widths of which can be varied from 10 nm up to several μm, depending on processing conditions. Combined with *in situ* nanoindentation in the transmission electron microscope (TEM), these films allow the investigation of nanoscale geometric confinement on dislocation-mediated plasticity. In the present study, np-Au films with a range of thicknesses were indented in the TEM. Measurements of indenter load versus displacement exhibit clear load drops, at intervals on the same length scale as the pore diameter. Although np-Au often exhibits macroscopic cracking, *in situ* TEM imaging clearly shows that individual np-Au ligaments are ductile, and that dislocations are generated even in 10 nm wide ligaments. These and other observations will be discussed in light of additional studies on the microstructure and mechanical behavior of np-Au.

## 11:00-11:20

### Observation of dislocation-grain boundary interactions in martensitic steel through in-situ nanoindentation in a TEM

**T. Ohmura[1], A.M. Minor[2], K. Tsuzaki[1] and J.W. Morris, Jr.[3]**
[1]*National Institute for Materials Science, Japan*
[2]*Lawrence Berkeley National Laboratory, CA*
[3]*University of California Berkeley, CA*

Dislocation-interface interactions in Fe-0.4wt%C tempered martensitic steel were studied through in-situ nanoindentation in a TEM. Two types of boundaries were imaged in the dislocated martensitic structure: a low-angle (probable) lath boundary and a coherent, high-angle (probable) block boundary. In the case of a low-angle grain boundary, the dislocations induced by the indenter piled up against the boundary. As the indenter penetrated further, a critical stress appears to have been reached and a high density of dislocations was suddenly emitted on the far side of the grain boundary into the adjacent grain. In the case of the high-

angle grain boundary, the numerous dislocations that were produced by the indentation were simply absorbed into the boundary, with no indication of pile-up or the transmission of strain. This surprising observation is interpreted on the basis of the crystallography of the block boundary.

## 11:20-11:40

### In situ TEM nanoindentation studies of alpha-$Al_2O_3$ and $Ti_3SiC_2$

**L. Johnson, F. Giuliani, L. Hultman**
*Thin Film Physics Division, Department of Physics Chemistry and Biology, IFM, Linkoping University*

Deformation of bulk sapphire and $Ti_3SiC_2$ thin film have been observed by in situ nanoindentation which have been made within a TEM. Samples were prepared by FIB milling. Indents made in sapphire show plasticity, and in particular basal slip. The maximum load achieved was 400 uN. Indentation in $Ti_3SiC_2$ showed a dislocation flow along the basal plane expanding into the film away from the indenter. Closer observation after the indentation showed kinks on both sides of the indent. The maximum load achieved as 430 uN. Movies of the deformations will be presented. Requirements placed on sample preparation and equipment by hard samples are discussed.

## 11:40-12:00

### On the Determination of Spherical Nanoindentation Stress-Strain Curves and Their Importance

**Sandip Basu, Alex Moseson and Michel Barsoum**
*Department of Materials Science and Engineering*
*Drexel University, Philadelphia, PA 19104*

Instrumented nanoindentation experiments, especially with sharp tips, are a well-established technique to measure the hardness and moduli values of a wide range of materials. However, and despite the fact that they can accurately delineate the onset of the elasto-plastic transition of solids, spherical nanoindentation experiments are less common. In this talk we outline a technique by which we combine, i) the results of continuous stiffness measurements with spherical indenters – with radii of 1 μm and/or 13.5 μm, ii) Hertzian theory and, iii) Vickers microhardness and Berkovich nanoindentations, to convert load/depth of indentation curves to their corresponding stress-strain curves. We applied the technique to fused silica, aluminum, iron and single crystals of sapphire, ZnO, GaN, mica and $LiNbO_3$, among others. In all cases, the resulting stress-strain curves obtained clearly delineated the elastic-to-plastic transition, i.e. the onset of yield, the hardening rates past the yield points, and, as important, steady state or minimal hardness values that were comparable to the Vickers microhardness values obtained

on the *same* surfaces. Furthermore, when both the 1 μm and 13.5 μm indenters were used on the same material, for the most part, the stress-strain curves traced a single trajectory. More recently we developed, and will report on, an algorithm to accurately and automatically determine the 'zero-point' of contact correction – a long-standing problem in nanoindentation. The versatility of the method and its importance will be discussed.

# Friday, August 3 – Afternoon

## INVITED 1:00-1:30

### In-situ electrical characterization during nanoindentation in silicon

**S. Ruffell, K. Sears, N. Fujisawa, J. E. Bradby and J. S. Williams**
*Department of Electronic Materials Engineering, Research School of Physical Sciences & Engineering, Australian National University, Canberra, 0200, Australia*

Nanoindentation-induced phase changes in silicon have attracted substantial interest over the last few decades. Although the phase transformations have been well studied, the mechanisms that govern the process are not totally understood. Furthermore, the electrical properties of the end phases of the residual nanoindents are unexplored. During loading, crystalline Si-I transforms to a metallic phase Si-II. On subsequent unloading, this further transforms to either amorphous silicon (a-Si) or a mixture of polycrystalline high pressure phases, Si-III and Si-XII depending on unloading conditions. We use in-situ electrical characterization to study the sequence of phase transformations during nanoindentation by utilizing the vastly differing electrical properties of the different phases of silicon (high pressure crystalline phases are orders of magnitude more conductive than a-Si). The in-situ electrical measurements have proven to be extremely sensitive to the structure of the nanoindented silicon both during and post-indentation. For example, we have shown that in a region in which only a-Si is expected to be formed during unloading, small volumes of Si-III and Si-XII can be detected through the electrical measurements. Using the nanoECR system as an electrical point probe we are able to investigate the end phases created by both micro- and nano-scale indentation. Variations within an indented zone can be probed with <100 nm resolution. Finally, in-situ electrical measurements are correlated with load/unload data, Raman micro-spectroscopy, and ex-situ electrical measurements.

## 1:30-1:50

### Multi-scale measurement of contact forces and current with a custom adhesion apparatus

**Dylan Morris[1], Doo-In Kim[1], Pradeep Namboodiri[1], Jaroslaw Grobelny[1,2], Robert Cook[1]**
[1]*National Institute of Standards and Technology, Materials Science and Engineering Laboratory, Ceramics Division, Nanomechanical Properties Group, Gaithersburg, MD*
[2]*University of Lodz, 90-236 Lodz, Poland*

Current measurements through a conducting probe may yield information about the surface condition, contacted area and junction behavior of two contacting materials. Conducting atomic force microscopy (C-AFM) and conducting-probe nanoindentation have both been established

as *in-situ* nanomechanical techniques. However, AFM cannot measure forces between macroscopic bodies, and typical nanoindenters cannot sense interaction forces such as those associated with snap-on and pull-off instabilities.

This talk will describe developmental work in conducting-probe force measurement with the NIST adhesion apparatus (NAA). The NAA is a custom instrument that utilizes a variable-length optical fiber cantilever. The optical fiber also acts as a very sensitive force-sensing element – the free end deflection of the cantilever is measured by coupling a laser diode to the fiber. The NAA may be used to measure forces between macroscopic bodies with the force sensitivity of an AFM. As well, the NAA also can apply and measure forces and displacements in the range of many nanoindentation instruments by simple adjustment of the cantilever length, which can be done rapidly without disturbing the sample or probe. Issues particular to current measurement with the NAA will be also be discussed, and results from C-AFM and conductance measurements with the NAA will be compared.

## 1:50-2:10

### Conductive Nanoindentation: In- situ Correlation of Mechanical Properties, Deformation Behavior, and Electrical Characteristics of Materials

**Ryan C. Major, David J. Vodnick, Syed Asif**
*Hysitron, Inc., 10025 Valley View Road, Minneapolis, MN 55344*

As materials and device development continues to proceed toward ever-decreasing length scales, successful integration of these materials and devices depends heavily on the ability to quantitatively assess and tailor their electrical and mechanical properties. Conductive nanoindentation combines nanoindenter hardware with a conductive probe and a voltage/current SourceMeter to get a time- based correlation of force, displacement, voltage, and current. The nanomechanical and electrical measurements used in tandem has proven to be highly sensitive to probe/sample contact conditions as well as material deformation behavior. This technique greatly enhances the information one can obtain from nanoscale point measurements. This presentation will cover the basic conductive nanoindentation technique along with intriguing results obtained from relatively mature materials, such as gold and metallic glasses to more advanced materials such as conductive polymers and conductive metal oxides (ITO).

## INVITED 2:10-2:40

### Application of Time-resolved Transmission Electron Microscopy to *In situ* Deformation Studies

**Thomas LaGrange[1], Andrew M. Minor[2], Geoffrey H. Campbell[1], Bryan W. Reed[1], Nigel D. Browning[1,3] and Wayne E. King[1]**

[1]*Chemistry, Materials and Life Sciences Directorate, Lawrence Livermore National Laboratory, Livermore, CA 94550, USA*

[2]*National Center For Electron Microscopy, Lawrence Berkeley National Laboratory, Berkeley, CA 94720, USA*

[3]*Department of Chemical Engineering and Materials Science, University of California, Davis, Davis, CA 95616, USA*

Understanding microscopic deformation mechanisms, i.e. dislocation dynamics, grain boundary motion, stress-induced phase transformations, etc., aid in the prediction of a material's macroscopic response under applied loads. With its high spatial resolution, *in situ* transmission electron microscopy deformation experiments have provided invaluable insight into these mechanisms. However, the standard video rate time-resolution of these observations limits the amount of information that can be inferred from the individual frames. In most cases, the important transient dynamics occur between the frames and are too rapid to be captured with these techniques. A recent example would be the *in situ* nanoindentation experiments on nanocrystalline Al by Minor *et al.,* where one frame shows a pristine Al grain and the next frame show the grain filled with dislocations. To capture the transient events in rapid materials' processes, the time resolution of *in situ* TEM must be increased to μs and ns levels. To meet this need, we have constructed a nanosecond time-resolved 'dynamic' TEM or DTEM at LLNL.

The DTEM currently generates single-shot images and diffraction patterns. "Single-shot" means that sufficient electrons for a complete image are present in a single electron pulse. The DTEM gains its high time and spatial resolution by marrying two nanosecond pulsed lasers to electron optics of a transmission electron microscope. One drives the photocathode (which replaces the standard thermionic cathode) to produce the brief electron pulse which has a bunch length proportional to the laser pulse duration. The other strikes the sample, initiating the process to be studied. In the DTEM's current mode of operation, a series of pump-probe experiments with varying time delays are needed to reconstruct the sequence of events in the material, i.e., evolution in microstructure in a phase transformation. In order to study, for example dislocation dynamics, new ways for triggering transient responses in materials using the DTEM single-shot approach must be developed.

This presentation will discuss recent developments and research on the DTEM and paths forward for *in situ* nanoindentation and deformation experiments. These experiments will also be discussed in the context of the physical and technical limitations of maintaining high spatial resolution at high temporal resolutions using the single-shot approach. The latter part of the talk will address the instrument design needed to push the time resolution to the picosecond regime, which is ultimate goal, observing material dynamics occurring at the speed

of sound.

Work performed under the auspices of the U.S. Department of Energy by the University of California, Lawrence Livermore National Laboratory and supported by the Office of Science, Office of Basic Energy Sciences, Division of Materials Sciences and Engineering, of the U.S. Department of Energy under contract No. W-7405-Eng-48.
UCRL-ABS- 231698

## 2:40-3:00

### An Environmental Stage for the Dynamic TEM: In Situ Microstructural Evolution in Varied Atmosphere at Nanosecond Scales

**Mitra L. Taheri[1], Pushkarraj Deshmukh[2], Paul E. Fischione[2], and Nigel D. Browning[1,3]**
[1]*Lawrence Livermore National Laboratory, Livermore, CA*
[2]*Fischione Instruments, Export, PA*
[3]*University of California-Davis, Davis, CA*

When studied using conventional in-situ TEM methods, events crucial to the understanding of the physical phenomena during various catalytic processes are missed due to the inability to capture them using video framing (30Hz). We present the development of an in-situ TEM technique to study thermally-activated and catalytic processes at nanosecond time scales using a unique environmental stage. The stage allows for gas input and laser heating; it also accommodates both a pulsed electron beam and a sample drive laser (to initiate a reaction). Our studies will focus on atomic shifts during sintering, atomic absorption and nanowire/nanotube catalyst growth interfaces. Imaging of catalyst materials under varied conditions related to oxidation will show how the structures change in reactive environments and determine the energetically preferred surface modifications. These critical events are able to be viewed at nanosecond temporal resolution and nanoscale spatial resolution in the DTEM using pulsed ultrafast electron beam imaging. The results will thus be critical to the growing need for alternative energy sources (i.e., a catalyst's role in fuel cells for energy) and various electronics such as blue light emitting diodes. These experiments could lead to future studies of the plastic deformation in nanostructured materials in gaseous environments using ultrafast TEM.

This work was performed under the auspices of the U.S. Department of Energy by the University of California, Lawrence Livermore National Laboratory and supported by the Office of Science, Office of Basic Energy Sciences, Division of Materials Sciences and Engineering, of the U.S. Department of Energy under Contract No. W-7405-Eng-48.

# ABSTRACTS – POSTER PRESENTATIONS

**(Poster abstracts submitted in time for publication)**

## Thursday, August 2
## 4:30-5:30

### Deformation Mechanics through Mechanical Probing inside a Transmission Electron Microscope

**M.S. Bobji**
*Department of Mechanical Engineering*
*Indian Institute of Science*
*Bangalore 560 012, India*

Transmission electron microscopy (TEM) has been extensively to study the deformation behaviour of materials. The ability of the TEM to look through the material coupled with its higher resolution has been used to decipher the deformation mechanisms of a wide variety of materials. Most of the TEM observations are post-mortem in the sense that the microstructural changes of the deformation frozen in the materials are analyzed in the TEM long after the application of the deforming forces and displacements.

Materials can be mechanically probed with indentation by applying localised forces and displacements. Localisation of the induced stresses means that the plasticity can be induced and controlled with precision in small volume. We have developed a TEM holder that can induce and control the deformation in situ with the help of a piezoelectric translator. The deformation event can be observed at very high resolution in real time and simultaneously the resistance offered by the material to the deformation can be measured. It is demonstrated that with this technique we can nucleate and track the movement of a single dislocation in polycrystalline aluminium.

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### In vitro nanoindentation of hyaline and repair cartilage and the influence of storage media on the mechanical properties

**O. Franke[1], V. Maier[1], K. Gelse[2], K. Durst[1], M. Göken[1]**
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[2]*Nikolaus Fiebiger Zentrum, University Erlangen, Glückstr. 6, 91054 Erlangen, Germany*

Cartilage is a very important component of all human joints which not only spreads the load on the subchondral bone but also provides lubrication for a higher mobility. Cartilage damage is not easily repaired and in many cases transplants offer the only chance to regain some of the original mobility affected joint. But only a limited number of joints can be replaced by implants

which furthermore exhibit a limited life-time. Because of these difficulties there are many approaches in medicine for developing a method to recover as much of the healthy hyaline cartilage as possible. In this work periosteal cells obtained thru a minor surgery from the shin were used as progenitor cells for cartilage repair. To study their chondrogenetic potential under stimulation BMP-2 was used and compared to unstimulated cells after 6 and 26 weeks implantation time. As a reference healthy hyaline cartilage was chosen. In vitro nanoindentation in a phosphate buffered saline (PBS) was used to assess the quality of the repair cartilage in comparison to the reference. structure The mechanical properties such as contact stiffness, modulus and hardness were measured and chosen as the mechanical parameters to evaluate the success of the tissue repair . Furthermore the effect of paraformaldehyde (PFA), which is a common method in histology for the storage of samples, was evaluated. It could be shown that the crosslinking of collagen fibers caused by PFA leads to a homogenization of the cartilage (Fig 1). Hence it should not be used to assess the mechanical properties of biomaterials by nanoindentation. To study the mechanical properties as close as possible to the in vivo conditions no fixation was used and the sample were immediately transferred into PBS and measured within 24 hours after extraction, ensuring that no microstructural changes (such as e.g. cross-links, decay,…) influenced the results.

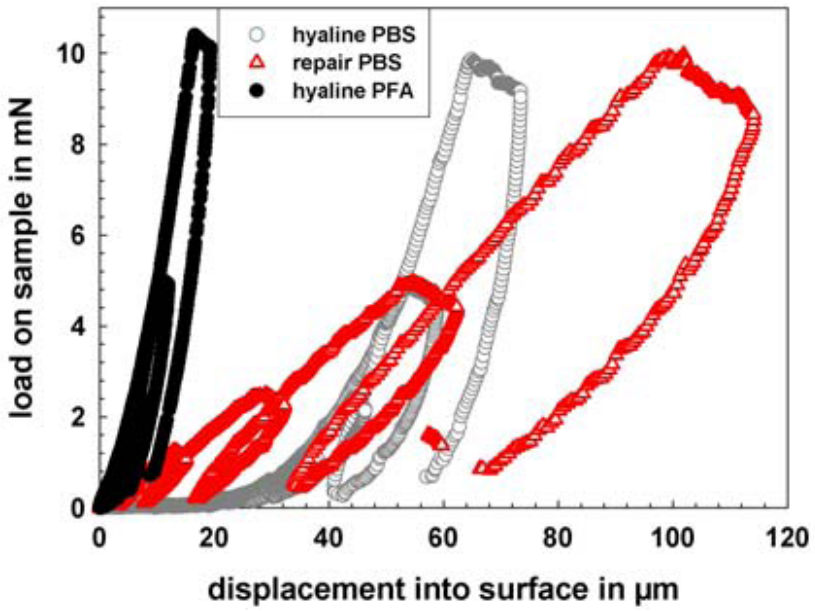


Fig 1: a) load-displacment curves for hyaline cartilage in PBS and PFA and repair cartilage without stimulation after 26 weeks

Nevertheless, it can be assumed that for even lower unloading rates the influence of time dependent effects on the contact stiffness will become significant especially at low applied loads. In this study the positive influence of the transgene stimulation could be shown after 6 weeks as well as after 6 months. Furthermore the comparison of the results from mechanical testing with histological analysis showed a good coincidence of the two methods. Hence nanoindentation is well suited for a quantitative description of the change in mechanical

properties caused by a replacement of collagen II fibers with the softer type I collagen found e.g. in fibrous cartilage.

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## On the Development of an In-situ SEM-Nanoindentation System

**Karolina Rzepiejewska–Malyska[1], Ryan C. Major[2], Edward Cyrankowski[2], Gerhard Buerki[1], Syed Asif[2] , Johann Michler[1]**

[1]*Materials Technology Department, Swiss Federal Laboratories for Materials Testing Research, EMPA, CH–3602 Thun, Switzerland*

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The decreasing scale of thin film coatings and nanoscale devices require new and innovative mechanical testing techniques. In-situ SEM-Nanoindentation is one technique that can provide a window to gain better understanding of the mechanical properties and material deformation behavior at an unprecedented scale. The current work summarizes our recent efforts to integrate depth-sensing nanoindentation with a high-resolution scanning electron microscope. The nanoindentation hardware provides for a high level of accuracy and precision in the application and measurement of load and displacement. The simultaneous high-resolution imaging allows for a more thorough and complete characterization of the material under test. This technique allows for the in-situ observation and detection of certain events such as crack initiation, pile-up and/or sink-in, and other material deformation phenomena. We will present some of our recent work on thin film coatings and bulk aluminum.

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## Nanoscale plasticity phenomena revealed through quantitative *in situ* TEM compression tests

**A.M. Minor[1], J. Ye[1], R.K. Mishra[2], Z.W. Shan[3], S.A. Syed Asif[3], O.L. Warren[3]**

[1]*National Center for Electron Microscopy, Lawrence Berkeley National Laboratory, Berkeley, CA*

[2]*General Motors Research and Development Center, Warren, MI*

[3]*Hysitron Incorporated, Minneapolis, MN*

The technique of micro-pillar compression tests is quickly becoming a new paradigm for small-scale mechanical testing, particularly for examining size effects at small length scales. Through quantitative *in situ* nano-compression tests in a transmission electron microscope (TEM) we can directly correlate the dynamic deformation mechanisms in submicron pillars with simultaneous measurement of the imposed stresses. This poster will demonstrate this capability through the *in situ* compression of single crystal pure Ni, and Al alloys. In the pure Ni pillars the phenomenon of mechanical annealing leads to dislocation-free structures that allow for direct comparison of the deformation behavior across samples with dramatically different dislocation densities. In the Al alloy systems the deformation can be more complicated, where cross-slip

can result in a three-dimensional dislocation network that leads to a rotation of the pillar structure under compression. These results will be shown in relation to the size effects seen in *ex situ* pillar compression tests where a direct relation between the yield stress and the diameter of pillar structures has been confirmed.

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## Ultrahigh strength and deformability of nanocrystalline hollow spheres

**Z.W. Shan[1,3,5], A. Cabot[2,3], A.M. Minor[1,3*], D.C. Chrzan[3,4], G. Adesso[2], M. Sherburne[4], S.A. Syed Asif[5], O.L. Warren[5], A.P. Alivisatos[2,3,4]**

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Nanocrystalline materials offer very high strength but are typically limited in their strain to failure, and efforts to improve deformability in these materials are usually found to be at the expense of strength. This work shows that nanocrystalline CdS synthesized into a spherical shell geometry can not only achieve ultra-high strength (approaching its ideal value), but also exhibit considerable deformation to failure (up to 20%) with a density as low as 50% when compared to a solid sphere. Using a combination of quantitative *in situ* compression in a transmission electron microscope (TEM) and finite element analysis (FEA), we show that the mechanical properties of nanoparticles can be directly measured and interpreted on an individual basis. By taking into account the structural hierarchy intrinisic to novel nanocrystalline materials such as this, we show it is possible to achieve both ultrahigh strength and deformability in a single nanoparticle.

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## In-situ compression testing of Al-Mo nanocomposite thin films

**Radmilovic V., Ye J., Lee Z., Minor A.M. and Dahmen U.**

*National Center for Electron Microscopy, LBNL, Berkeley, CA 94720, USA*

**Ophus C., Luber E. and Mitlin D.**

*Dept. of Materials and Chemical Eng., University of Alberta*

It has been shown previously that the addition of 32at.% of Mo to aluminum films leads to the precipitation of BCC Mo-rich particles in a metallic Al-Mo amorphous matrix, resulting in an increased nanoindentation hardness to a peak value of 6.3 GPa. In this study we investigated the deformation behavior of nanocomposite (Mo rich nanocrystals in Al rich amorphous matrix) Al-Mo binary alloy thin films using quantitative in-situ uniaxial compression testing of nanopillars in a JEOL 3010 transmission electron microscope. Examination of the recorded

video showed that the pillar yielded immediately upon contact with the flat diamond tip. After approximately 90 nm of displacement the formation of a shear band was observed and coincided with the sudden load drop. The observed apparent plastic strain is localized, indicating that the deformation mechanisms are directly related to nanoscale shear instabilities. As soon as the band has propagated across the pillar cross-section, it forms a kink at the pillar surface and suggests that failure is associated with shear band formation. This study demonstrates that in a sub-micron volume of amorphous materials under uniaxial compression the apparent plastic strain is localized in a very few shear bands.

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## Determination of nano-mechanical properties of lignocellulosic fibers by nanoindentation and its limitation

**Siqun Wang[1], Seung-Hwan Lee[1,2], Cheng Xing[1], George M. Pharr[3]**

[1]*Forest Product Center, Forestry, Wildlife and Fisheries, The University of Tennessee, TN, US*

[2]*Biomass Technology Research Center, National Institute of Advanced Industrial Science and Technology, Hiroshima, Japan*

[3]*Department of Materials Science and Engineering, The University of Tennessee, Knoxville, TN and Metals and Ceramic Division, Oak Ridge National Laboratory, Oak Ridge, TN, US*

This study has investigated the mechanical properties and creep behavior of lignocellulosic fibers on nanoscale depth by using continuous stiffness technique. In the fiber-reinforced polymer composites, the mechanical properties of the final composite depend on the mechanical properties of reinforcing materials. Therefore, a better understanding of the properties and characteristics of reinforcing raw material will be helpful to estimate the overall properties of fiber-reinforced composites materials and will provide the optimum design of composite materials. In particular, compared to conventional tensile creep tests, the continuous nanoindentation creep experiments are particularly useful as they simulate creep resulting from asperity contact. The nanoindentation gives a direct measure of mean stress and contact stiffness, and being insensitive to drift, allows the accurate observation of creep in small indents to be carried out over a long time period. Lyocell fibers with different tensile modulus and wood fibers refined under different steam pressure were used in this study. Two different kinds of nano-scale values for hardness and elastic modulus, mean value from 150nm to 300 nm depth, and the value from final unloading, were obtained and showed that there was no significant difference in two values. The hardness and elastic modulus values in longitudinal direction were higher than those in transversal direction. In both directions, the values for lyocell fiber with a higher tensile modulus were higher than those for lyocell fiber with a lower tensile modulus. The continuous nanoindentation for creep behavior of lyocell fiber showed that the creep test can be used to study the stress relaxation, evolved in an indentation experiment at room temperature. Based on finite element analysis, we will discuss effects of neighboring material properties on the nanoindentation measurement.

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# An Advantage of Displacement-Controlled Nanoindentation of Polymers

**Oden L. Warren[1], Wei Che[1], Michael J. Fasolka[2], Christopher M. Stafford[2]**

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Nanoindentation of polymers has been problematic because the standard unloading analysis assumes a wholly elastic rather than viscoelastic unloading process. In load-controlled nanoindentation experiments, the polymer's creep response often causes a forward-going nose over the initial portion of the unloading curve, which, if present, yields an initial unloading slope of the wrong sign. Invoking an extended hold period at the maximum load to reduce the creep rate is a common approach to at least achieving an initial unloading slope of the proper sign. However, we have determined that invoking a peak-interaction hold period is not necessary to yield constant nanoindentation results over a wide range of deformation rates providing the experiment is performed under infrequently used displacement control. A sensible explanation will be given for this conclusion made from our multi-parameter combinatorial nanoindentation study performed on thickness-gradient polystyrene libraries having narrow molecular weight distribution.

# Workshop Participants

| Name | Affiliation | Contribution |
|---|---|---|
| Alsem, Daan Hein | Lawrence Berkeley National Lab | Speaker |
| Armbruster, Barbara | JEOL | Attendee |
| Asif, Syed | Hysitron, Inc. | Speaker |
| Askari, Davood | Univesrity of Hawaii at Manoa | Attendee |
| Balk, John | University of Kentucky | Invited speaker |
| Barsoum, Michel | Drexel University | Speaker |
| Basu, Sandip | Drexel University | Attendee |
| Bobji, M.S. | India Institute of Science | Poster |
| Brooks, Iain | Integran Technologies Inc. | Attendee |
| Cheng, Ming-Chieh | National Tsing Hua University, Taiwan | Attendee |
| Chrzan, Daryl | University of California, Berkeley | Invited speaker |
| Chung, Hsiu-Ying | UCLA | Attendee |
| Dahmen, Ulrich | Lawrence Berkeley National Lab | Attendee |
| Delplancke-Ogletree, Marie-Paule | Lawrence Berkeley National Lab | Attendee |
| Du, Ming Liang | Goodyear Tire & Rubber Co. | Attendee |
| Ebenstein, Donna | Bucknell University | Speaker |
| El-Deiry, Paul | Schick-Wilkinson Sword | Attendee |
| Fonseca, Luis | University of Puerto Rico | Attendee |
| Franke, Oliver | University of Erlangen, Germany | Poster |
| Haque, Aman | Penn State University | Invited speaker |
| Hartfield, Cheryl | Omniprobe, Inc. | Attendee |
| Hornberger, Lee | BAE Systems | Attendee |
| Houston, Jack | Sandia National Labs | Speaker |
| Hsu, Richard | UCLA | Attendee |
| Huang, Jianyu | Sandia National Labs | Speaker |
| Jaroenapibal, Papot | University of Pennsylvania | Attendee |
| Jen, James | Vistakon, Johnson & Johnson | Attendee |
| Johnson, Lars | Linköping University, Sweden | Speaker |
| Kim, Suhan | Lawrence Berkeley National Lab | Attendee |
| Kinney, Christopher | University of California, Berkeley | Attendee |
| Klein, René | Huntsman Polyurethanes, Belgium | Attendee |
| Kraft, Oliver | Forschungszentrum Karlsruhe, Germany | Attendee |
| Kwok, Chi Kong | Komag | Attendee |
| LaGrange, Thomas | Lawrence Livermore National Lab | Invited speaker |
| Li, Biyun | UCLA | Attendee |
| Lou, Jun | Rice University | Attendee |
| Lowry, Matthew | University of California, Berkeley | Attendee |
| Magid, Karen | University of California, Berkeley | Attendee |
| Major, Ryan | Hysitron, Inc. | Speaker, Poster |
| Mao, Scott | University of Pittsburgh | Speaker |
| Mara, Nathan | Los Alamos National Lab | Speaker |
| Marks, Laurence | Northwestern University | Speaker |
| Milas, Mirko | Brookhaven National Lab | Attendee |
| Minor, Andrew | Lawrence Berkeley National Lab | Organizer, Poster |

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|---|---|---|
| Mishra, Raj | General Motors | Attendee |
| M'ndange-Pfupfu, Ariel | Northwestern University | Attendee |
| Morris, Bill | University of California, Berkeley | Attendee |
| Morris, Dylan | National Institute of Standards and Technology | Speaker |
| Mossman, Michele | Ball Corporation | Attendee |
| Nie, Xueyuan | University of Windsor, Canada | Attendee |
| Nix, William | Stanford University | Plenary speaker |
| Ogletree, Frank | Lawrence Berkeley National Lab | Attendee |
| Ohmura, Takahito | National Institute for Materials Science, Japan | Speaker |
| Prakash, Vikas | Case Western Reserve University | Speaker |
| Radmilovic, Velimir | Lawrence Berkeley National Lab | Poster |
| Resto, Oscar | University of Puerto Rico | Attendee |
| Ringnalda, Jan | FEI Co. | Invited speaker |
| Ruffell, Simon | Australian National University | Invited speaker |
| Ruoff, Rod | University of Texas at Austin | Speaker |
| Shan, Zhiwei | Hysitron, Inc. | Speaker |
| Sherburne, Matt | University of California, Berkeley | Attendee |
| Smith, Douglas | National Institute of Standards and Technology | Attendee |
| Solá, Francisco | University of Puerto Rico | Attendee |
| Sriram, Vinay | UCLA | Attendee |
| Taheri, Mitra | Lawrence Livermore National Lab | Speaker |
| Tong, Michael | UCLA | Attendee |
| Tran, Lloyd | California Institute of Nanotechnology | Attendee |
| Van Swygenhoven, Helena | Paul Scherrer Institute, Switzerland | Keynote speaker |
| Varnum, Dan | Oxford Instruments, UK | Attendee |
| Wahl, Kathryn | Naval Research Lab | Invited speaker |
| Wang, Siqun | University of Tennessee | Poster |
| Warren, Oden | Hysitron, Inc. | Organizer, Poster |
| Withey, Elizabeth | University of California, Berkeley | Attendee |
| Wu, Junqiao | University of California, Berkeley | Attendee |
| Wyrobek, Thomas | Hysitron, Inc. | Attendee |
| Xu, Guanghai | Intel Corp. | Attendee |
| Yaglioglu, Onnik | FormFactor Inc. | Attendee |
| Ye, Jia | Lawrence Berkeley National Lab | Speaker |
| Yim, Joanne | University of California, Berkeley | Attendee |
| Zhang, Xiao Feng | Hitachi High Technologies America | Attendee |